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SUBSTITUTE SPECIFICATION

SPECTROPHOTOMETER

FIELD OF THE INVENTION

[0001] The present invention relates to a system for analyzing the spectral absorption of a material in a test sample, and in particular to improvements in the system of the type using detection apparatus.

BACKGROUND OF THE INVENTION

[0002] The simplest spectroscope splits incident visible light into spectral lines that can be observed by the human eye. In more complicated analysis, for example spectrochemical analysis, the substance under investigation is heated, so that it emits radiation. Each component of the substance emits a characteristic radiation, and this can be used as a means of identification. The radiation is passed through a diffraction grating or a prism to separate it into its constituent wavelengths. Detectors are then used to observe or record details of the spectrum, and an instrument can be used to measure the wavelengths and intensities of spectral lines. A permanent record of the results (a spectrograph) may be made to allow more detailed analysis. Comparison of the spectrum with the spectra of known, pure, substances allows the components to be identified and, with quantitative analysis, their relative proportions determined. This offers an extremely sensitive method of analysis of chemical substances, and automated spectroscopic procedures are now used routinely in laboratories.

[0003] Most laboratory apparatus that are currently used for the measurement of the concentration of a material in a solution are relatively complex in nature. Their degree of complexity is at least partially a cause for several disadvantages. Firstly, they are relatively expensive. Secondly, they are often relatively delicate as they use prisms and dispersion gratings and are generally unsuitable for use in the field or in normal manufacturing and processing environments. And thirdly, they are generally specific in purpose and often cannot be readily adapted for other applications.

[0004] The term "material" shall be used in its broadest sense and shall not be restricted to solid matter but also to liquids or gases. In addition the term "solution" shall also be taken to include the gaseous phase.

[0005] International patent application number WO96/31764 discloses a method and apparatus for the quantitative determination of particles in fluid. This apparatus comprises one or more light emitters, and one or more light detectors sensitive to the output of the emitters. Data is gathered from a plurality of signal paths between the emitter and detector. This data is subsequently evaluated by comparison with known data for different fluid particles in a fluid sample.

[0006] United States Patent 4,158,505 describes a spectrophotometer consisting of a wideband light source, paths provided for the sample and reference lights, and a chopper wheel allowing the sample and reference light to be interspersed with periods of blocked light and to be alternately incident on a dispersion grating and thus transmitted onto a linear array of photodiodes.

[0007] United States Patent 3,955,082 describes a single photodetector for measuring a variety of wavelengths. The single photodetector is constructed from a plurality of detector sections each having a variable bandwidth and controlled by varying the reverse bias voltage.

[0008] United States Patent 5,357,343 describes a spectrophotometer consisting of a single emitter detector and a rotating chopper. The rotary chopper contains filters to select wavelengths to be incident on the detector at any one time. All the inventions described in each specification possess many of the general disadvantages described previously.

SUMMARY OF THE INVENTION

[0009] It is therefore an object of the present invention to provide a spectrophotometer which overcomes the above-mentioned disadvantages in the prior art or which will at least provide the industry with a useful choice.

[0010] Accordingly, in a first aspect the present invention consists in an analyzer or spectrophotometer for the detection of material in a sample comprising:

a source adapted to direct radiation at least at the sample, the radiation incident on or reflected by the sample able to be varied,

a detector for detecting at least radiation reflected by the sample, wherein the detector has a spectral response able to be varied and an output depending on radiation incident thereon and the spectral response, and

a controller or processor receiving the output, configured or programmed to:

vary the intensity of the source,

vary the spectral response of the detector, and

determine a characteristic of the sample based on the output in relation to the variations.

[0011] In a second aspect the present invention consists in an analyzer or spectrophotometer for the detection of material in a sample comprising:

means for directing radiation at the sample,

means for varying the radiation incident on or reflected by the sample,

means for detecting at least radiation reflected by the sample,

means for varying the spectral response of the means for detecting,

means for providing a output representative of the detected reflected radiation, and

means for determining a characteristic of the sample based on the output in relation to the variations.

[0012] In a third aspect the present invention consists in a method of detecting material in a sample comprising the steps of:

directing radiation at the sample,

varying the radiation incident on or reflected by the sample,

detecting at least radiation reflected by the sample using a detector,

varying the spectral response of the detector,

providing a variable spectral response output representative of the detected radiation, and

determining a characteristic of the sample based on the output in relation to the variations, wherein

the radiation directed at the sample is varied by varying the voltage or current supplied to the radiation source,

the radiation directed at the sample is varied by varying the transmission path between the radiation source and the sample,

the transmission path is varied by varying the size of the aperture through which radiation is directed at the sample,

the size of the aperture is varied by a rotating wheel with different sized apertures through which radiation is directed at the sample,

the radiation directed at the sample is varied by the variation in intensity when switching the radiant source on or by pulsing the radiation source,

the radiation reflected by the sample is detected by a photodiode and the spectral response of the output is varied by varying the width of the depletion zone within the diode,

the width of the depletion zone within the diode is varied by varying the reverse voltage applied across the diode and the output being the resulting current,

the output signal from the detector is amplified and digitized prior to being supplied to the controller,

the controller is a microprocessor,

the detector is a photodiode detector,

the source is a light emitting diode,

the source is a tungsten filament lamp, and

the source is a gas discharge lamp.

[0013] In a fourth aspect the present invention consists in an analyzer or spectrophotometer for the detection of material in a sample comprising:

a source adapted to direct radiation at least at the sample,

a detector configured to provide an output indicative of at least radiation reflected by the sample,

a variable transmission path for radiation between the source, the sample or the detector, configured to vary at least intensity of radiation incident on the sample, and

a controller or processor receiving the output and operating the transmission path, configured or programmed to:

determine the radiation reflected from the source off the sample,

determine the radiation directly from the source, and

determine a characteristic of the sample based on the output in relation to variations in the transmission path.

[0014] In a fifth aspect the present invention consists in an analyzer or spectrophotometer for the detection of material in a sample comprising:

means for directing radiation at at least the sample,

means for varying the intensity of the radiation directed at the sample,

means for detecting at least radiation reflected by the sample,

means for detecting at least radiation directly from the source, and

means for determining a characteristic of the sample based on the reflected radiation and the direct radiation, in relation to the variations in intensity.

[0015] In a sixth aspect the present invention consists in a method of detecting material in a sample comprising the steps of:

directing radiation at at least the sample,

varying the intensity of the directed radiation,

detecting at least radiation reflected by the sample,

detecting at least radiation directly from the source, and

determining a characteristic of the sample based on the reflected radiation in relation to the direct radiation, wherein

the radiation reflects off the sample along a sample path and radiation passes directly to the detector along a reference path,

a blocking member has at least 3 cyclic modes:

a first mode during which the radiation passes the reference path,

a second mode during which the radiation passes the sample path, and

a third mode during which the radiation is blocked,

the blocking member is rotatable about a central axis,

during the first mode the intensity of the radiation through the sample path is varied,

the intensity is varied by providing different sized apertures in an annular path through the blocking member, and

the blocking member includes indexing and a sensor(s) detects the position of the blocking member.

[0016] To those skilled in the art to which the invention relates, many changes in construction and widely differing embodiments and applications of the invention will suggest themselves without departing from the scope of the invention as defined in the claims. The disclosures and the descriptions herein are purely illustrative and are not intended to be in any sense limiting.

[0017] The invention consists in the foregoing and also envisions construction of which the following gives examples.

BRIEF DESCRIPTION OF THE DRAWINGS

[0018] The embodiment of the present invention will now be described with reference to the accompanying drawings in which:

[0019] **Figure 1** shows a block diagram of the overall structure of the present invention;

[0020] **Figure 2a** shows a cross-sectional view of the spectral analyzing apparatus of the present invention;

[0021] **Figure 2b** shows a detail drawing of the reflection angle through the sample being tested;

[0022] **Figure 2c** shows a detail drawing of the chopper wheel in the beam splitting apparatus;

[0023] **Figure 3a** shows a cross-sectional drawing of an alternate embodiment of the spectral analyzing apparatus;

[0024] **Figure 3b** shows a plan cross-sectional drawing of the alternate embodiment of the spectral analyzing apparatus;

[0025] **Figure 3c** is a section view of the chopping wheel of the alternate embodiment of the spectral analyzing apparatus;

[0026] **Figure 4a** illustrates how the photodetector is penetrated by short wavelength photons;

[0027] **Figure 4b** illustrates how the photodetector is penetrated by long wavelength photons;

[0028] **Figure 5a** shows the response of a typical photodetector by varying the intensity of the light source;

[0029] **Figure 5b** shows the response of a typical photodetector by varying the reverse voltage across the photodetector;

[0030] **Figure 5c** shows the response of a typical photodetector by varying both the light intensity and the reverse voltage;

[0031] **Figure 6** is a section view through an alternative chopping wheel; and

[0032] **Figure 7** is an exploded view of an alternative chopping wheel.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0033] One embodiment of the present invention relates to a spectrophotometer for providing a qualitative and quantitative measure of material size, density and spectral response of a sample. In particular, the spectrophotometer reads the reflectance of a sample. In an alternate embodiment, the spectrophotometer reads the absorbance of a sample. One embodiment of the apparatus of the present invention will be comprised of at least one emitter and at least one detector. The emitter may be a single light emitter or alternately be comprised of an array of several light emitters that function as if a single light emitter. The detector may be a standard-type photodiode capable of measuring a spectrum of frequencies commensurate with the application. The emitter and detector may be controlled by a microprocessor and/or in turn are connected to an external PC.

[0034] Referring to Figure 1, the light emitter 1 of the present invention generates radiation of a spectrum commensurate with the application. This may be over a broad range of frequencies including visible and infrared regions of the electromagnetic spectrum, a broadband signal, or a narrow band signal over a small range. Inexpensive emitters such as light emitting diodes, gas discharge lamps or tungsten filament lamps are examples. The detector 9 may for example be a photodiode to detect incident light signals.

[0035] The emitter 1 and detector 9 are both controlled by a controller module 3, for example a microprocessor. Microprocessor 3 includes software, control algorithms or component logic to achieve two things:

i) variation in the intensity of light reflected by the sample,

ii) variation of the spectral response of the detector.

Radiation Source

[0036] There are a number of ways of doing both but for simplicity only a few examples will be discussed here. For example the microprocessor 3 could control the intensity of the light emitted from the emitter 1 by varying the current supply 2. The operation of the detector 9 is controlled by varying the supply of voltage or reverse bias 10. Any signal generated by the detector 9 is amplified and converted into a digital format before being processed by the microprocessor 3.

[0037] In another embodiment, the light from the emitter 1 is split up into separate paths before falling incident on the detector 9 seen in Figure 2c. This operation is performed by a device called a beam splitter or a chopping wheel 5. The rotation of the chopping wheel 5 may be controlled by the microprocessor 3 so that at any one time the detector 9 will only sense one signal. Typically the chopping wheel 5 blocks the path of other signals while allowing one signal to pass and fall incident on the detector 9. The three signals possible in this embodiment are the reference signal, the sample signal and the dark signal (no signal).

[0038] Figure 2a illustrates the beam splitting apparatus 30 of the present invention. The beam splitting apparatus 30 is comprised of several elements. These include a light source 32, a chopping wheel 33, paths to direct the lights 40, 42 and 44, a detector 35, and a sample under test 31. The sample under test is typically contained by a test tube 31. The test tube 31 may be comprised of two sections with different diameters: 31a and 31b. The lower section 31b of this test tube 31 has a smaller diameter than the top section 31a to allow for small amounts of a sample to be tested. Alternately a standard test tube may be used, this test tube having a same diameter for its entire length. The test tube 31 containing the sample is inserted into a holding cavity 50 within the spectrophotometer for measurement.

[0039] Figure 2b illustrates the reflection angle of the light from the light source 32 towards the chopper wheel 33, along path 44. The angle of reflection is substantially 90 degrees so that the reflected light, travelling along path 40, is perpendicular to the detector 35. This allows for optimum information transfer onto the light detector 35 with a minimum of diffraction distortion.

[0040] With reference to Figure 2c, the chopper wheel is comprised of a circular-type barrel with one section of the wheel being substantially flat as shown in 34. The depth of this flat section 34 is dictated by the diameter of the light transmission pathways 40, 42 and 44. The depth of the flat section 34 on wheel 33 may be substantially the same as the diameter of the light transmission pathways 40, 42 and 44. Alternately, flat section 34 is approximately half way between the circular edge and the center of rotation of the chopper wheel 33.

[0041] One complete revolution of the chopper wheel 33 will result in any one of three signals at the detector 35. These three signals include a reference signal, a sample signal, and a dark signal. The reference signal is when point 33a on the chopper wheel 33 is at substantially positive 10 degrees from the vertical axis. The information provided at the detector 35 is coming directly from the light source 32. The sample signal is detected when point 33b on the chopper wheel 33 is at substantially negative 10 degrees from the vertical axis. This information at the detector 35 is comprised of the radiation coming from the reflectance off the sample. Any other time when there is no light incident on the detector 35 is known as the dark current. Dark current is important as it provides information for the calibration of the spectrophotometer. This information generally relates to the temperature drift of the detector 35 and the associated electronics.

[0042] An alternative embodiment of the physical structure of the spectrophotometer is shown in Figures 3a to 3c. Referring to Figure 3a, the angle between the light source 51 and detector 55 as it reflects off sample 50 is approximately 45 degrees. The chopping wheel 56 dictates when the light signal will be incident on the detector 55. In Figure 3c, the chopping wheel 53 is divided into three sections, namely a hole section 62, a mirror section 60 and a black section 61. The hole section 62 allows the signal to reflect off the sample 50

and be detected by the detector 55. The mirror section 60 reflects the signal directly to the detector 55. This results in reference readings. The black section 61 stops the transmission of light signal to the detector 55 so dark current readings may be taken. In this alternative embodiment, the chopping wheel 53 is off to one side of the sample 50. This allows transmission of the signal light through one section of the chopping wheel 53 at any one time, so simplifying the control of the chopping wheel 53.

[0043] In a further improvement seen in Figures 6 and 7, the chopper wheel 600 may provide two functions, firstly varying the intensity of the light source reflected off the sample, as well as providing an intermittent direct path to the detector. This embodiment includes a wheel 600 driven by motor 602 with a number of apertures of varying diameter 704 which allow radiation from the source 606 to reflect off the sample 608 to the detector 610. These match up with apertures 705 in front of the detector 610 which don't vary in size. Wheel 600 also includes an axial rim 612 which blocks light directly from the source to the detector, except for a notch 714 to intermittently allow a direct path between the source and the detector and block the sample for calibration purposes. There are also a number of apertures around the periphery 716 and two sensors 718 in the base 620 to allow the microprocessor to calculate the position of the wheel such that the intensity of the light can be recorded alongside the signal received from the emitter. The moving parts are held into the base 620 by insert 622. Sample 608 is held in position by spring 624.

[0044] In a still further improvement, variation of the intensity of the emitter could be achieved as the light source changes intensity over a very short period of time. For example when a filament is first turned on, the amount of light emitted is not immediately at its maximum and takes a finite period of time to increase, which depends on the type of filament and the ambient temperature. In order to detect both the intensity of light generated and the consequent signal received by the detector, different methods would need to be employed. For example, intensity of the light generated could be detected by a high resolution measurement of the voltage drop directly over the filament, giving indication of the change in intensity of the radiation generated. Alternatively the source could be pulsed.

Radiation Detector

[0045] The detector 35 of the present invention is typically of a *pn*-junction or a *p-i-n* photodiode type. *P-i-n* junction photodiodes would provide the fast response necessary if high resolution encoding of incident radiation is required, e.g. intensity changes, as the source is turned on or pulsed. Referring to Figures 4a and 4b, the photodetector 35 is connected in reverse bias with a DC voltage source 68; in particular the negative terminal is connected to the *p*-side 65 of the diode 35 and the positive terminal is connected to the *n*-side 67 of the diode 35.

[0046] The operation of the detector 35 as it relates to the present invention will now be explained. When a photon of light is absorbed by the detector 35, it excites an electron and produces a single pair of charge carriers, an electron and a hole, where a hole is simply the absence of an electron in the diodes semiconductor lattice. Current passes through the semiconductor when the charge carriers separate and move in opposite directions. The detector 35 collects the photon-induced charge carriers that can be measured as current or voltage at its electrodes.

[0047] An *n*-type semiconductor material 67 may be doped with Silicon or Germanium to produce an excess of electrons, whereas a *p*-type material 65 has an excess of holes, or an electron deficiency. The area where these two materials meet is called the *pn*-junction. At the *pn*-junction, this disparity creates a concentration gradient that causes electrons to diffuse into the *p*-layer and holes to diffuse into the *n*-layer. This diffusion results in an opposing electrical potential, often referred to as an internal bias. Charge carriers cannot reside in this region, therefore it is termed the depletion region.

[0048] In detector 35 of the present invention, light enters the device through a thin *p*-type layer. Absorption causes light intensity to drop exponentially with penetration depth. Any photons absorbed near the depletion region produce charge carriers that are immediately separated and swept across the *pn*-junction by the inherent internal bias of the device. Charge carriers created outside the depletion region will move randomly, many of them eventually entering the depletion region to be swept rapidly across the *pn*-junction. Some of them will recombine and disappear without ever reaching the depletion region. This movement of charge carriers across the *pn*-junction upsets the electrical balance and

produces a small photocurrent that is detected at the electrodes of the detector. The electrical current or voltage produced is proportional to the light intensity incident on the detector 35.

[0049] Figure 5a illustrates the response 72 of a typical detector 35 to a varying intensity 71 for a signal. It can be seen that varying the intensity 71 of the light source will affect the bandwidth or the total response 73 of the detector 35. In particular by increasing the intensity 71 of the light source to high level 91, the range of wavelengths 73 that can be measured at a particular time is decreased. Reducing the intensity 71 of the light source to low level 90 increases the range of wavelengths 73 that can be measured but only up to the maximum bandwidth response of the detector 72.

[0050] By increasing the intensity 71 of the light source 1, the Quantum efficiency of the detector is increased. The Quantum efficiency is defined as the ratio of the photocurrent in electrons to the incident light intensity in photons (or the sensitivity of the photodetectors to different wavelengths).

[0051] Referring to Figure 4a, short wavelengths 80 of light penetrate a short distance into the structure of the detector 35; i.e. light will interact close to the surface of the diode. Referring to Figure 4b, longer wavelengths 81 of light penetrate deeper into the structure of the detector 35, or in extreme cases, the detector 35 becomes totally transparent to long wavelengths 81. Short wavelengths 80 of light are comprised of high energy photons while longer wavelengths 81 contain lower energy photons. The detector 35 only produces a current or voltage at its electrodes if the photons absorbed have enough energy or are close enough to traverse the *pn*-junction. This effect is called the "cutoff wavelength".

[0052] Photons with a wavelength less than the cutoff and in close proximity with the *pn*-junction will produce current or voltage. Photons with a longer wavelength greater than the cutoff will not produce current or voltage.

[0053] In order to control the cutoff wavelength it is desirable to control the thickness of the depletion region 66. One example way to expand this layer 66 is to apply an external electrical bias (voltage) 68. By applying an external electrical bias 68, the *p*-type 65 and *n*-type 67 regions reduce in thickness, so reducing the efficiency of the longer wavelengths 81

in creating charge carriers. The thickness of these layers is directly controlled by the magnitude of external electrical bias 68. The greater the magnitude of external electrical bias 68, the thinner the regions 65 and 67, where charge carriers are formed, and the smaller the cutoff wavelength is. Ideally the control of the voltage 68 is provided by the microprocessor 3. One skilled in the art will appreciate other ways of varying the spectral response of the detector.

[0054] Figure 5b illustrates the incorporation of the external bias variable 68 to aid in the control of the detector 35. This external bias variable 68 is the magnitude of the external voltage 68 applied to the detector 35. As previously discussed, varying the voltage 68 varies the thickness of the regions 65 and 67 where charge carriers are formed, so effecting the response bandwidth of the detector 35. Specifically, by increasing the magnitude of the external voltage supply 68, the upper bandwidth response 93 of the diode decreases. The control of external voltage 68 may be controlled by the microprocessor 3. Voltage 68 applied to the detector 35 may be changed in a stepwise manner. Alternately the external voltage 68 is changed in a continuous fashion.

[0055] By combining the control of the light source intensity 71 and the external voltage 68 applied to the detector 35 by the microcontroller 3, individual components from the sample signal can be determined. In practice, for every stepped change in the external voltage 68, a number of different intensities 71 are emitted from the light source 1. For every choice of cutoff band 70, a new range of detectable spectra is observed. The band sweep (due to bias voltage) and intensity sweep (due to light source) lead to a combined set of data points arranged across the full spectrum of the device under investigation. This way the characterization of all the spectra under investigation is possible.

[0056] There are numerous possible embodiments in the process of analyzing the spectral absorption of a material in a test sample. In one embodiment, a signal, for example the reference signal, is transmitted through the chopper wheel 33 to the detector 35. The controlling module, typically a microprocessor 3, selects a voltage to be supplied to the detector 35, controlling the width of the depletion region 66, and thus selects a predetermined bandwidth that the detector 35 will be sensitive to. The microprocessor 3 then varies the voltage supplied to the light source 1, thus varying the intensity of the

broadband light signal. The detector 35 will send representative signals to the microprocessor 3. This data is then stored in the microprocessor until the test is finished. The changes in the level of voltage supplied to the detector 35 select detection bandwidths. This process repeats until measurements have been performed at all preselected bandwidths. The entire process is repeated again for the next light signal, for example the light signal from the sample path.

[0057] In another embodiment of the present invention, one signal, for example the reference signal, is transmitted through the chopping wheel to the detector. The microprocessor selects the predetermined bandwidth that the detector is sensitive to, performed by selecting the voltage level supplied to the detector. The microprocessor 3 then varies the voltage level supplied to the light source, thus varying the intensity of the light signal. The detector generates representative signals that are transmitted to the microprocessor. The chopper wheel then rotates and blocks the reference signal and allows the next signal to be transmitted, for example the sample signal. Again the microprocessor varies the intensity of the light source and the measured values are stored in the microprocessor. As the chopper wheel rotates again there is a dark interval. This is known as the dark current. There is no light incident on the detector but an inherent current will flow across the *pn*-junction of the photodiode. This current level is measured and used by the microprocessor to calibrate for any temperature drift in the electronics.

[0058] The output signals from detectors are in the form of voltages; these are measured from the detectors terminals. These signals are representative of the light incident on the detectors surface. The output signals are small; they are proportional to the amount of current flowing through the diode as a result of light being detected. These signals are too small and are in the wrong format to be accurately detected by the microprocessor 3, so they are modified by output circuitry 6. This output circuitry 6 is comprised of two sections, namely an amplification section 7 and a conversion section 8. The amplification section 7 is comprised of an operational amplifier circuit. The gain provided by this circuit is dependent on the components used, therefore gain may be increased or decreased accordingly. Alternately other types of amplification circuit 7 may be used in a similar manner. Once the signal is amplified, it is converted from an analogue signal to a digital signal. This is performed either by a dedicated analogue-to-digital converter circuit 8 or in

an analogue-to-digital converter contained within the microprocessor 3. The output signals from the detector are now in a format that the microprocessor 3 can identify and use.

[0059] Signals from the detector are continuously supplied to the microprocessor 3 as it is continuously measuring light incident on its surface. The microprocessor 3 takes discrete measurements from the continuously supplied signal and stores these signals in its memory.

[0060] The output of the detector reflects the presence of material in a sample. Once the test is completed, the microprocessor 3 stores the measured values in an array in its memory.

Data Analysis

[0061] Subsequent evaluation of these measured values may be made by a number of methods. Some trials and experimentation may be relied upon to determine the best method for obtaining values indicative of material presence in a sample. However, for ease of use, most embodiments will rely upon the comparison of received measured values with collected or stored data. This data may be values which have been pre-programmed into the microprocessor 3 so that the subsequent collection of initial set-up data by the user may not be required. This stored data may be comprised of values typical for the type of samples to be analyzed, although it is envisioned for most embodiments that there will be provided provision for routine calibration using reference samples either to check accuracy and/or to adjust the apparatus. Calibration information will be stored in the microprocessor 3 or in software being run by the external processing means. The use of software may be more flexible, allowing for the updating of software to change the performance of the apparatus. In addition, calibration data is updated whenever a new calibration is run.

[0062] After treatment of all calibration data with a multiple regression method, the correlation factor and the intercept or the free factor are obtained. Summation of this formula results in typically eight locations in the total calibration data array. These eight readings relate to the locations that provided the best measurements.

[0063] The spectral response, material size and density is obtained by the multiplication of each individual sample reading at the predetermined position in the array with its regression coefficient factor and addition of the free factor as shown in the following formula:

$$\text{Result} = \text{free factor} + \sum_{n=1}^{8} \text{coef}_n \cdot \text{meas}_n$$